Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tris(*N*-acetylglycinato-κ^2O,O')triaquasamarium(III)

Prashant M. Kameshwar,[a] Amey Wadawale[b] and Vishnu R. Ajgaonkar[a]*

[a]Department of Chemistry, University of Mumbai, Vidyanagari, Mumbai 400 098, India, and [b]Chemistry Division, Bhabha Atomic Research Centre, Trombay, Mumbai 400 085, India
Correspondence e-mail: vrajgaonkar@yahoo.com

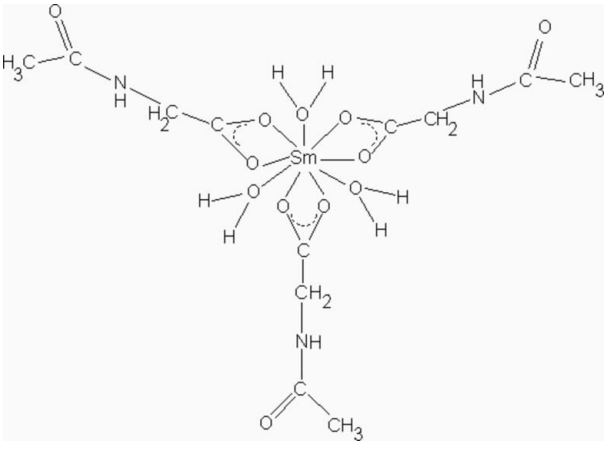
Received 14 August 2007; accepted 21 September 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; R factor = 0.020; wR factor = 0.051; data-to-parameter ratio = 8.4.

The title complex, $[Sm(C_4H_6NO_3)_3(H_2O)_3]$, was prepared by reacting samarium(III) carbonate with *N*-acetylglycine in an aqueous medium. The Sm^{III} atom is coordinated by nine O atoms, six of them belonging to the three carboxylate groups of the ligands and three to the water molecules. The coordination geometry can be described in terms of a 4,4,4-tricapped triangular prism. The molecule lies on a threefold rotation axis.

Related literature

For related compounds, see: Kamath & Udupa (1983); Kameshwar *et al.* (2007); Udupa & Krebs (1978); Zeng & Pan (1992).



Experimental

Crystal data

$[Sm(C_4H_6NO_3)_3(H_2O)_3]$
M_r = 552.69
Trigonal, $R3$
a = 16.580 (4) Å
c = 5.978 (1) Å
V = 1423.2 (5) Å^3
Z = 3
Mo $K\alpha$ radiation
μ = 3.16 mm^{-1}
T = 298 (2) K
0.40 × 0.20 × 0.20 mm

Data collection

Rigaku AFC-7*S* diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.364, T_{max} = 0.570
(expected range = 0.339–0.531)
1396 measured reflections
900 independent reflections
702 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
3 standard reflections
every 150 reflections
intensity decay: 0.6%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.051
S = 1.07
900 reflections
107 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.81 e Å^{-3}
$\Delta\rho_{min}$ = −1.21 e Å^{-3}
Absolute structure: Flack (1983), with 176 Friedel pairs
Flack parameter: −0.03 (2)

Table 1
Selected geometric parameters (Å, °).

O2—Sm1	2.538 (4)	O4—Sm1	2.403 (4)
O1—Sm1	2.505 (4)		
O4—Sm1—O4^{i}	78.2 (2)	O4—Sm1—O2	159.32 (14)
O4—Sm1—O1	142.61 (16)	O4^{i}—Sm1—O2	93.23 (15)
O4^{i}—Sm1—O1	80.61 (16)	O4^{ii}—Sm1—O2	118.80 (14)
O4^{ii}—Sm1—O1	67.44 (15)	O1—Sm1—O2	51.42 (12)

Symmetry codes: (i) $-y+2, x-y+1, z$; (ii) $-x+y+1, -x+2, z$.

Data collection: *WinAFC* (Rigaku/MSC, 2004); cell refinement: *WinAFC*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors are grateful to Dr Vimal K. Jain, Head of the Synthesis and Pure Materials Section, Chemistry Division, Bhabha Atomic Research Centre, for providing the single-crystal X-ray diffraction facility and for helpful comments. PMK gratefully acknowledges financial support from the University of Mumbai.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2078).

References

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Kamath, P. & Udupa, M. R. (1983). *Indian J. Chem. Sect. A*, **22**, 218–220.
Kameshwar, P. M., Wadawale, A. & Ajgaonkar, V. R. (2007). *Acta Cryst.* E**63**, m2584.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Rigaku/MSC (2004). *WinAFC* (Version 1.0.5) and *CrystalStructure* (Version 3.7.0). Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Udupa, M. R. & Krebs, B. (1978). *Inorg. Chim. Acta*, **31**, 251–255.
Zeng, H. & Pan, K. (1992). *Jiegou Huaxue*, **11**, 388–392.

  doi:10.1107/S1600536807046429

supplementary materials

Acta Cryst. (2007). E**63**, m2598 [**doi:10.1107/S1600536807046429**]

Tris(*N*-acetylglycinato-$\kappa^2 O,O'$)triaquasamarium(III)

P. M. Kameshwar, A. Wadawale and V. R. Ajgaonkar

Comment

Rare earth complexes of *N*-acetylglycine were synthesized and reported to be isostructural and hexagonal (Kamath & Udupa, 1983). However, the detailed structural analysis was not given. The crystal structures of neodymium, europium and erbium complexes of *N*-acetylglycine have been reported (Zeng & Pan, 1992). The compounds were found to be isostructural and trigonal.

In the title compound, the Sm^{III} atom, lying on a threefold rotation axis, is coordinated by six O atoms from three carboxylate groups and three O atoms from three water molecules (Fig. 1). The three chelated carboxylate rings are completely staggered. The three Sm—O(water) bonds are also completely staggered with the same angle of 78.2 (2)° between two such bonds (Table 1). The angle O1—C1—O2 is 120.8 (6)°, while the angle subtended at Sm by the carboxylate O atoms (O1—Sm1—O2) is 51.4 (1)°. The bond distances between the two carboxylate O atoms and the Sm atom differ by only 0.033 Å. The bond lengths of the two carboxylate O atoms to the C atom are almost identical. The carboxylate group is thus resonance stabilized and functions symmetrically as a bidentate chelate. Apart from the carboxylate group, the bond distances and bond angles of *N*-acetylglycinate moiety in the title compound are not significantly different from those of free *N*-acetylglycine and its copper (Udupa & Krebs, 1978), neodymium, europium and erbium complexes (Zeng & Pan, 1992).

The title compound is isostructural with its terbium (Kameshwar *et al.*, 2007), neodymium, europium and erbium analogues (Zeng & Pan, 1992). The coordination geometry in the title compound can be described in terms of a 4,4,4-tricapped triangular prism.

Experimental

The title compound was synthesized by adding samarium carbonate (0.376 g, 2.5 mmol) to *N*-acetylglycine (0.878 g, 7.5 mmol) dissolved in 50 ml water and allowing to react on a steam bath till the carbonate dissolved. A few mg of the carbonate were added to ensure that no unreacted acid was present. The unreacted carbonate was filtered off and the filtrate was evaporated naturally at ambient temperature. The crystals suitable for X-ray diffraction were picked up and dried in air. Analysis, calculated for $C_{12}H_{24}N_3O_{12}Sm$: C 26.08, H 4.38, N 7.60, Sm 27.20%; found: C 25.89, H 4.31, N 7.42, Sm 26.91%.

Refinement

H atoms of the methyl group were positioned geometrically and refined as riding atoms, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C). The other H atoms were found in a difference Fourier map and refined isotropically.

Figures

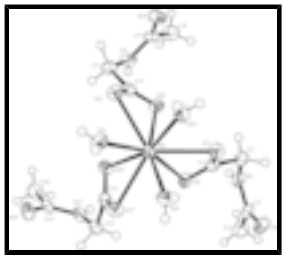


Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) 2 − *y*, 1 + *x*-*y*, *z*; (ii) 1 − *x* + *y*, 2 − *x*, *z*.]

Tris(*N*-acetylglycinato-κ^2*O*,*O*')triaquasamarium(III)

Crystal data

$[Sm(C_4H_6NO_3)_3(H_2O)_3]$	$Z = 3$
$M_r = 552.69$	$F_{000} = 825$
	$D_x = 1.935$ Mg m^{-3}
Trigonal, *R*3	$D_m = 1.932$ Mg m^{-3}
	D_m measured by flotation method
	Mo *K*α radiation
Hall symbol: R 3	λ = 0.71069 Å
a = 16.580 (4) Å	Cell parameters from 25 reflections
b = 16.580 (4) Å	θ = 12.7–16.7º
c = 5.978 (1) Å	μ = 3.16 mm^{-1}
α = 90º	*T* = 298 (2) K
β = 90º	Needle, colourless
γ = 120º	0.40 × 0.20 × 0.20 mm
V = 1423.2 (5) Å^3	

Data collection

Rigaku AFC-7S diffractometer	$R_{int} = 0.029$
Radiation source: fine-focus sealed tube	$\theta_{max} = 27.5$º
Monochromator: graphite	$\theta_{min} = 3.7$º
T = 298(2) K	*h* = −21→18
ω/2θ scans	*k* = 0→21
Absorption correction: ψ scan (North *et al.*, 1968)	*l* = −7→4
$T_{min} = 0.364$, $T_{max} = 0.570$	3 standard reflections
1396 measured reflections	every 150 reflections
900 independent reflections	intensity decay: 0.6%
702 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.020$	$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.051$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.07$	$\Delta\rho_{max} = 0.81$ e Å^{-3}
900 reflections	$\Delta\rho_{min} = -1.21$ e Å^{-3}
107 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
3 restraints	Extinction coefficient: 0.0017 (4)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with 176 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: −0.03 (2)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C4	0.7757 (5)	0.6593 (4)	−0.1291 (10)	0.0392 (13)
H4C	0.7402	0.5934	−0.1548	0.059*
H4A	0.8165	0.6890	−0.2538	0.059*
H4B	0.7343	0.6836	−0.1124	0.059*
O2	0.9222 (3)	0.8784 (3)	0.3629 (7)	0.0286 (8)
O1	0.9998 (3)	0.8499 (3)	0.6168 (6)	0.0296 (8)
C1	0.9635 (6)	0.8361 (5)	0.4255 (11)	0.0244 (14)
O4	1.0216 (3)	1.1147 (4)	0.9427 (7)	0.0309 (9)
H1	0.938 (6)	0.771 (5)	−0.046 (13)	0.05 (2)*
H2A	0.968 (5)	0.716 (5)	0.371 (11)	0.035 (17)*
H2B	1.039 (5)	0.808 (4)	0.214 (10)	0.029 (16)*
H2W	0.997 (8)	1.109 (9)	1.064 (10)	0.11 (5)*
H1W	1.051 (7)	1.1708 (19)	0.921 (17)	0.04 (3)*
Sm1	1.0000	1.0000	0.6673	0.01945 (14)
N1	0.9221 (3)	0.7447 (3)	0.0689 (8)	0.0284 (10)
O3	0.7980 (3)	0.6347 (3)	0.2547 (7)	0.0371 (10)
C3	0.8321 (4)	0.6779 (3)	0.0787 (9)	0.0285 (11)
C2	0.9797 (4)	0.7734 (4)	0.2680 (9)	0.0282 (10)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C4	0.046 (3)	0.037 (3)	0.031 (3)	0.018 (3)	−0.009 (3)	−0.004 (2)
O2	0.034 (2)	0.0271 (19)	0.0256 (19)	0.0162 (18)	−0.0032 (17)	0.0001 (15)
O1	0.039 (2)	0.032 (2)	0.0205 (19)	0.0195 (17)	−0.0054 (15)	−0.0001 (15)
C1	0.025 (3)	0.022 (3)	0.021 (3)	0.009 (2)	0.002 (2)	0.004 (2)

O4	0.037 (2)	0.028 (2)	0.022 (2)	0.011 (2)	0.0082 (18)	−0.0022 (19)
Sm1	0.02189 (15)	0.02189 (15)	0.01455 (18)	0.01095 (8)	0.000	0.000
N1	0.039 (3)	0.022 (2)	0.018 (2)	0.0109 (19)	0.0003 (18)	−0.0006 (17)
O3	0.041 (2)	0.030 (2)	0.030 (2)	0.0095 (18)	0.0049 (18)	0.0062 (16)
C3	0.037 (3)	0.017 (2)	0.028 (3)	0.011 (2)	0.000 (2)	−0.0023 (19)
C2	0.034 (3)	0.024 (2)	0.025 (2)	0.013 (2)	0.002 (2)	−0.001 (2)

Geometric parameters (Å, °)

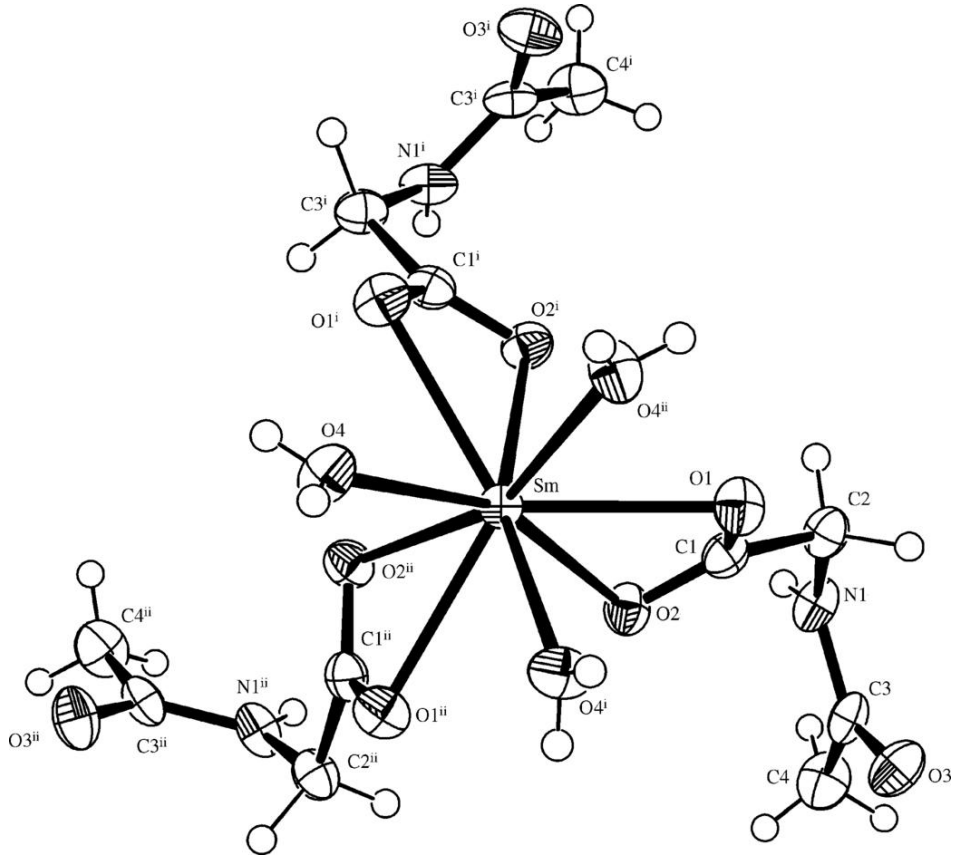
C4—C3	1.491 (8)	Sm1—O4^{ii}	2.403 (4)
C4—H4C	0.9600	Sm1—O1^{i}	2.505 (4)
C4—H4A	0.9600	Sm1—O1^{ii}	2.505 (4)
C4—H4B	0.9600	Sm1—O2^{ii}	2.538 (4)
O2—C1	1.258 (8)	Sm1—O2^{i}	2.538 (4)
O2—Sm1	2.538 (4)	Sm1—C1^{ii}	2.863 (8)
O1—C1	1.258 (9)	Sm1—C1^{i}	2.863 (8)
O1—Sm1	2.505 (4)	N1—C3	1.342 (7)
C1—C2	1.522 (9)	N1—C2	1.450 (7)
C1—Sm1	2.863 (8)	N1—H1	0.79 (7)
O4—Sm1	2.403 (4)	O3—C3	1.239 (6)
O4—H2W	0.81 (8)	C2—H2A	1.06 (7)
O4—H1W	0.82 (2)	C2—H2B	0.91 (6)
Sm1—O4^{i}	2.403 (4)		
C3—C4—H4C	109.5	O1^{i}—Sm1—O2^{i}	51.42 (12)
C3—C4—H4A	109.5	O1^{ii}—Sm1—O2^{i}	124.04 (13)
H4C—C4—H4A	109.5	O1—Sm1—O2^{i}	78.72 (14)
C3—C4—H4B	109.5	O2—Sm1—O2^{i}	74.26 (14)
H4C—C4—H4B	109.5	O2^{ii}—Sm1—O2^{i}	74.26 (14)
H4A—C4—H4B	109.5	O4—Sm1—C1	166.99 (15)
C1—O2—Sm1	91.6 (4)	O4^{i}—Sm1—C1	90.7 (2)
C1—O1—Sm1	93.1 (4)	O4^{ii}—Sm1—C1	92.91 (19)
O2—C1—O1	120.8 (6)	O1^{i}—Sm1—C1	120.94 (17)
O2—C1—C2	121.9 (6)	O1^{ii}—Sm1—C1	101.65 (19)
O1—C1—C2	117.1 (6)	O1—Sm1—C1	26.03 (17)
O2—C1—Sm1	62.4 (4)	O2—Sm1—C1	26.05 (18)
O1—C1—Sm1	60.9 (3)	O2^{ii}—Sm1—C1	98.17 (17)
C2—C1—Sm1	159.2 (5)	O2^{i}—Sm1—C1	70.74 (18)
Sm1—O4—H2W	131 (9)	O4—Sm1—C1^{ii}	90.7 (2)
Sm1—O4—H1W	124 (7)	O4^{i}—Sm1—C1^{ii}	92.9 (2)
H2W—O4—H1W	104 (10)	O4^{ii}—Sm1—C1^{ii}	166.99 (15)
O4—Sm1—O4^{i}	78.2 (2)	O1^{i}—Sm1—C1^{ii}	101.65 (19)
O4—Sm1—O4^{ii}	78.2 (2)	O1^{ii}—Sm1—C1^{ii}	26.03 (17)
O4^{i}—Sm1—O4^{ii}	78.2 (2)	O1—Sm1—C1^{ii}	120.94 (17)
O4—Sm1—O1^{i}	67.44 (15)	O2—Sm1—C1^{ii}	70.74 (18)

O4^{i}—Sm1—O1^{i}	142.61 (16)	O2^{ii}—Sm1—C1^{ii}	26.05 (18)
O4^{ii}—Sm1—O1^{i}	80.61 (16)	O2^{i}—Sm1—C1^{ii}	98.17 (17)
O4—Sm1—O1^{ii}	80.61 (16)	C1—Sm1—C1^{ii}	96.75 (18)
O4^{i}—Sm1—O1^{ii}	67.44 (15)	O4—Sm1—C1^{i}	92.9 (2)
O4^{ii}—Sm1—O1^{ii}	142.61 (16)	O4^{i}—Sm1—C1^{i}	166.99 (16)
O1^{i}—Sm1—O1^{ii}	118.57 (4)	O4^{ii}—Sm1—C1^{i}	90.7 (2)
O4—Sm1—O1	142.61 (16)	O1^{i}—Sm1—C1^{i}	26.03 (17)
O4^{i}—Sm1—O1	80.61 (16)	O1^{ii}—Sm1—C1^{i}	120.94 (17)
O4^{ii}—Sm1—O1	67.44 (15)	O1—Sm1—C1^{i}	101.65 (19)
O1^{i}—Sm1—O1	118.57 (4)	O2—Sm1—C1^{i}	98.17 (17)
O1^{ii}—Sm1—O1	118.57 (4)	O2^{ii}—Sm1—C1^{i}	70.74 (18)
O4—Sm1—O2	159.32 (14)	O2^{i}—Sm1—C1^{i}	26.05 (18)
O4^{i}—Sm1—O2	93.23 (15)	C1—Sm1—C1^{i}	96.75 (18)
O4^{ii}—Sm1—O2	118.80 (14)	C1^{ii}—Sm1—C1^{i}	96.75 (18)
O1^{i}—Sm1—O2	124.04 (13)	C3—N1—C2	120.9 (5)
O1^{ii}—Sm1—O2	78.72 (14)	C3—N1—H1	115 (6)
O1—Sm1—O2	51.42 (12)	C2—N1—H1	123 (6)
O4—Sm1—O2^{ii}	93.23 (15)	O3—C3—N1	120.8 (5)
O4^{i}—Sm1—O2^{ii}	118.80 (14)	O3—C3—C4	122.0 (5)
O4^{ii}—Sm1—O2^{ii}	159.32 (14)	N1—C3—C4	117.2 (5)
O1^{i}—Sm1—O2^{ii}	78.72 (14)	N1—C2—C1	114.8 (5)
O1^{ii}—Sm1—O2^{ii}	51.42 (12)	N1—C2—H2A	112 (4)
O1—Sm1—O2^{ii}	124.04 (13)	C1—C2—H2A	103 (4)
O2—Sm1—O2^{ii}	74.26 (14)	N1—C2—H2B	104 (4)
O4—Sm1—O2^{i}	118.80 (14)	C1—C2—H2B	107 (4)
O4^{i}—Sm1—O2^{i}	159.32 (14)	H2A—C2—H2B	115 (6)
O4^{ii}—Sm1—O2^{i}	93.23 (15)		
Sm1—O2—C1—O1	17.9 (7)	O1—C1—Sm1—O4^{ii}	11.7 (4)
Sm1—O2—C1—C2	−156.5 (6)	C2—C1—Sm1—O4^{ii}	−78.4 (13)
Sm1—O1—C1—O2	−18.1 (7)	O2—C1—Sm1—O1^{i}	−105.1 (4)
Sm1—O1—C1—C2	156.5 (5)	O1—C1—Sm1—O1^{i}	92.5 (3)
C1—O1—Sm1—O4	167.7 (4)	C2—C1—Sm1—O1^{i}	2.4 (14)
C1—O1—Sm1—O4^{i}	111.6 (5)	O2—C1—Sm1—O1^{ii}	28.8 (4)
C1—O1—Sm1—O4^{ii}	−167.4 (4)	O1—C1—Sm1—O1^{ii}	−133.7 (4)
C1—O1—Sm1—O1^{i}	−102.7 (4)	C2—C1—Sm1—O1^{ii}	136.2 (13)
C1—O1—Sm1—O1^{ii}	53.8 (4)	O2—C1—Sm1—O1	162.5 (7)
C1—O1—Sm1—O2	9.7 (4)	C2—C1—Sm1—O1	−90.1 (14)
C1—O1—Sm1—O2^{ii}	−7.0 (4)	O1—C1—Sm1—O2	−162.5 (7)
C1—O1—Sm1—O2^{i}	−69.0 (4)	C2—C1—Sm1—O2	107.4 (14)
C1—O1—Sm1—C1^{ii}	23.8 (5)	O2—C1—Sm1—O2^{ii}	−23.4 (4)
C1—O1—Sm1—C1^{i}	−81.5 (5)	O1—C1—Sm1—O2^{ii}	174.2 (4)

C1—O2—Sm1—O4	−149.6 (4)	C2—C1—Sm1—O2^{ii}	84.1 (13)
C1—O2—Sm1—O4^{i}	−85.0 (4)	O2—C1—Sm1—O2^{i}	−93.5 (5)
C1—O2—Sm1—O4^{ii}	−6.7 (5)	O1—C1—Sm1—O2^{i}	104.1 (4)
C1—O2—Sm1—O1^{i}	91.8 (4)	C2—C1—Sm1—O2^{i}	14.0 (13)
C1—O2—Sm1—O1^{ii}	−151.3 (4)	O2—C1—Sm1—C1^{ii}	2.9 (4)
C1—O2—Sm1—O1	−9.7 (4)	O1—C1—Sm1—C1^{ii}	−159.6 (4)
C1—O2—Sm1—O2^{ii}	155.9 (4)	C2—C1—Sm1—C1^{ii}	110.3 (12)
C1—O2—Sm1—O2^{i}	78.2 (5)	O2—C1—Sm1—C1^{i}	−94.8 (3)
C1—O2—Sm1—C1^{ii}	−177.0 (4)	O1—C1—Sm1—C1^{i}	102.8 (5)
C1—O2—Sm1—C1^{i}	88.7 (3)	C2—C1—Sm1—C1^{i}	12.7 (14)
O2—C1—Sm1—O4	127.5 (8)	C2—N1—C3—O3	−5.1 (8)
O1—C1—Sm1—O4	−34.9 (12)	C2—N1—C3—C4	174.2 (5)
C2—C1—Sm1—O4	−125.0 (12)	C3—N1—C2—C1	−76.6 (7)
O2—C1—Sm1—O4^{i}	95.9 (4)	O2—C1—C2—N1	−17.3 (9)
O1—C1—Sm1—O4^{i}	−66.6 (4)	O1—C1—C2—N1	168.2 (6)
C2—C1—Sm1—O4^{i}	−156.7 (13)	Sm1—C1—C2—N1	−112.9 (13)
O2—C1—Sm1—O4^{ii}	174.1 (5)		

Symmetry codes: (i) −*y*+2, *x*−*y*+1, *z*; (ii) −*x*+*y*+1, −*x*+2, *z*.

Fig. 1


O3i
C4i
C3i
N1i
C3i
C1i
O1i
O2i
O4ii
O4
Sm
O1
C2
C1
O2ii
O2
N1
C4ii
C1ii
N1ii
O1ii
O4i
C3
O3ii
C3ii
C2ii
C4
O3